

CLAIMS

1. A method for measuring ultralow permeation through a sample using a radioactive compound, which method comprises the steps of:

mounting a sample through which permeation is to be measured so as to provide controlled access to an upstream surface of the sample in a first chamber and to a downstream surface thereof in a second chamber, wherein said second downstream chamber has a volume of not greater than about 10 cm³,

supplying a radioactive gas from a source to be in contact with the upstream surface of the sample in the first chamber,

collecting radioactive gas permeating from the downstream surface of the sample by circulating a very slow flow of dry carrier gas at a rate of not greater than about 1.5 liter per hour through the second chamber to provide a radioactive stream,

flowing said radioactive stream from said second chamber to an entrance to an ionic chamber not greater than about 2 liters in volume containing a beta-particle radiation monitor,

continuously monitoring said stream for beta particle radioactivity and generating signals, and

receiving signals from said radiation monitor in conversion means and converting the signals to calculate the permeation rate through the sample at that moment, whereby the sensitivity of the method allows measurement of permeation of radioactive gaseous compounds through samples that have barrier properties which permit permeation at rates of only 0.0001 to about 0.00001 gm/sq.m/day or less.

2. The method for measuring permeation according to claim 1 wherein said radioactive gas is tritiated water vapor (HTO).

3. The method for measuring permeation according to claim 2 wherein a relative humidity of HTO between about 85% and 100% is supplied to the first chamber throughout the entire test period for the sample.

4. The method for measuring permeation according to claim 1 wherein said radioactive gas is HTO and said carrier gas is dry methane.

5. The method for measuring permeation according to claim 1 wherein said radioactive gas is carbon¹⁴ monoxide (¹⁴CO).

6. The method for measuring permeation according to claim 5 wherein said carrier gas is dry argon.

7. The method for measuring permeation according to claim 1 wherein said carrier gas enters said second chamber at a pressure just sufficient to maintain the desired very slow flow and is vented to the atmosphere through an absorption device which removes all of said radioactive compound from said carrier gas stream.

8. The method for measuring permeation according to claim 7 wherein said carrier gas enters at a pressure of not greater than about 1.1 atm.

9. The method for measuring permeation according to claim 1 wherein said sample is a polymeric film.

10. The method for measuring permeation according to claim 1 wherein said sample is an adhesive perimeter seal between two thin plates.

11. Apparatus for measuring ultralow permeation through a sample which apparatus comprises:

means for mounting the sample through which permeation is to be measured to provide controlled access to a first chamber in communication with an upstream surface of the sample and a second chamber at a downstream surface thereof, said second downstream chamber having a volume not greater than about 10 cm³,

means for supplying a radioactive gas to the first chamber where it will be in contact with the upstream surface of the sample,

means for circulating a very slow flow of carrier gas through the second chamber to provide a radioactive stream containing the gas permeating from the downstream surface of the sample, said second chamber having a volume not greater than about 10 cm³,

conduit means for flowing said stream from said second chamber to an ionic chamber that contains a radiation monitor for continuously monitoring said stream for beta particle radioactivity and for creating signals indicative of radioactivity, said ionic chamber having a volume not greater than about 2 liters, and

conversion means for receiving signals from said radiation monitor and converting the signals to calculate the permeation rate through the sample at that moment, whereby the sensitivity is such as to detect permeation of radioactive gaseous compounds through samples that have barrier properties which permit permeation at rates of only 0.0001 to about 0.00001 gm/m²/day or less.

12. The apparatus for measuring permeation according to claim 11 which further comprises a station for removing all radioactive gas from the carrier gas stream following its exit from said radiation chamber and for accumulating said removed gas.

13. The apparatus for measuring permeation according to claim 12 wherein there are also provided:

a ball valve for segregating said radioactive gas supply means from communication with said first chamber following testing of one sample, and

means for purging said first chamber with gas to remove all radioactive gas therefrom and for directing said purge gas into said removal station prior to venting of said purge gas.

14. The apparatus for measuring ultralow permeation according to claim 13 wherein said supply means provides a relative humidity of tritiated water vapor (HTO) between about 85% and 100% throughout the entire test period for a sample.

15. The apparatus for measuring permeation according to claim 11 wherein

said mounting means is designed to support a thin film composite that includes a polymeric layer.

16. The apparatus for measuring ultralow permeation according to claim 15 wherein said mounting means includes a third chamber which surrounds the periphery of said mounted film composite and wherein said carrier gas circulating means can circulate a slow flow of carrier gas through said third chamber.

17. The apparatus for measuring permeation according to claim 11 wherein said mounting means supports a first flat plate having an opening which extends therethrough and wherein a second flat plate having an area greater than said opening is affixed to a downstream surface of said first flat plate by line of adhesive of defined thickness and width which completely encircles said opening and creates a seal between said plates, whereby one can determine the thickness and width of said line of adhesive needed to create an adhesive perimeter seal which exhibits desired barrier properties to permeation therethrough.

18. The apparatus for measuring permeation according to claim 11 wherein said conversion means has the capability to present either an instantaneous rate of permeation or an average rate measured over a period of hours or days.

19. A method for testing a perimeter seal including adhesive material for ultralow permeation therethrough using a radioactive compound, which method comprises the steps of:

providing two plates, one of which has an opening therethrough which is spaced from the edges thereof, and assembling said plates so their facing surfaces are spaced substantially equidistant to each other by a continuous seal that includes adhesive material and encircles said opening,

mounting said assembly so as to provide controlled access to said plate containing the opening in a first chamber and to the other plate in a second chamber, supplying a radioactive gas to the first chamber from a source so as to

fill the region between said two plates and thus be in contact with an upstream surface of the continuous seal,

collecting radioactive gas permeating from the downstream surface of the perimeter seal by circulating a very slow flow of dry carrier gas at a rate of not greater than about 1.5 liters per hour through the second chamber to provide a collection stream,

flowing said radioactive collection stream from said second chamber to an entrance to an ionization chamber containing a beta-particle radiation monitor,

continuously monitoring said stream for beta-particle radioactivity and generating signals, and

receiving signals from said radiation monitor in conversion means and converting the signals to calculate the permeation rate through the mounted perimeter seal at that moment.

20. The method for testing for permeation according to claim 19 wherein said radioactive gas is either tritiated water vapor (HTO) or ^{14}CO .